

Department of Civil Engineering
College of Engineering and Technology
Old Dominion University
Norfolk, VA 23529

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POLYMER INFILTRATION STUDIES

By
Joseph M. Marchello, Principal Investigator

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Norman J. Johnston, Technical Monitor
MD-Polymeric Materials Branch

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P.O. Box 6369
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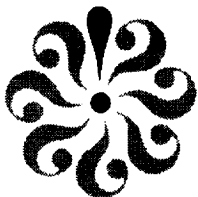
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Final Report

POLYMER INFILTRATION STUDIES

Summary

Significant progress was been made during the project on the preparation of carbon fiber composites using advanced polymer resins. The results were set forth in quarterly and annual reports and in publications and presentations at national and international meetings.

Among the research activities and achievements reported therein are:

- Assistance in acquisition of bench scale automated prepreg placement, ATP, equipment at NASA-LARC
- Participation in the design and operation of a Prepregger for solution and hot sled operation in making tape from polymer solutions and from powder coated tow
- Ribbonizing Powder-Impregnated Towpreg
- Experimentally determine short -time ribbon bonding kinetics for In-situ ATP.
- Powder Curtain Prepreg Process development.
- Textile applications of powder coated tow.

Polymer Infiltration Studies

Polymer infiltration investigations were directed toward development of methods by which to produce advanced composite material for automated part fabrication utilizing textile and robotic technology in the manufacture of subsonic and supersonic aircraft. This object was pursued through research investigations at NASA Langley Research Center and by stimulating technology transfer between contract researchers and the aircraft industry.

Project Results

The findings and results of the project are summarized in the following paper entitled " Powdered-Coated Towpreg: Avenues to Near Net Shape Fabrication of High performance Composite". This paper will be presented at the ICCM-10 Conference in Vancouver in August 1995.

Twenty-nine papers were written during the course of the grant. The are presented in the following list of publications. Copies of these papers were included in the appropriate quarterly and annual reports, and are available on request.

Two patents were issued during the grant and four additional patent applications have been submitted. Copies of the first patent was submitted earlier. A copy of the second one is attached. Copies of the patent applications were included in quarterly reports and are available on request.

ICC-M-10
Aug 1995

Vancouver

POWDER-COATED TOWPREG: AVENUES TO NEAR NET SHAPE FABRICATION OF HIGH PERFORMANCE COMPOSITES

N. J. Johnston and R. J. Cano
NASA Langley Research Center

J. M. Marchello
Old Dominion University
and

D. A. Sandusky
The College of William and Mary

ABSTRACT

Near net shape parts were fabricated from powder-coated preforms. Key issues including powder loss during weaving and tow/tow friction during braiding were addressed, respectively, by fusing the powder to the fiber prior to weaving and applying a water-based gel to the towpreg prior to braiding. A 4:1 debulking of a complex 3-D woven powder-coated preform was achieved in a single step utilizing expansion rubber molding. Also, a process was developed for using powder-coated towpreg to fabricate consolidated ribbon having good dimensional integrity and low voids. Such ribbon will be required for in situ fabrication of structural components via heated head advanced tow placement. To implement process control and ensure high quality ribbon, the ribbonizer heat transfer and pulling force were modeled from fundamental principles. Most of the new ribbons were fabricated from dry polyarylene ether and polyimide powders.

INTRODUCTION

In the last several years, powder impregnation of carbon fibers has been greatly refined at Langley Research Center to provide a low cost starting material for fabricating high performance composites from high melt viscosity difficult-to-process matrix materials [1].

Two paths are being pursued in composite part fabrication from powder-coated towpreg. The towpreg is being woven and braided into various textile preforms which are thermoform consolidated into a final part. Alternately, towpreg is being converted into a thin consolidated ribbon for lay-down in heated head automated tow placement machines.

TEXTILE APPLICATIONS

Compression or autoclave molding of textile preforms made from polymer powder-coated yarns offers an alternative to resin transfer molding for the fabrication of net-shape parts. Powder coating the fiber bundle prior to weaving or braiding requires special consideration for both

textile processing and for the subsequent debulking of the preforms [2]. Carbon fiber (6K and 12K AS4, Hercules) coated with epoxy thermoset powder (AMD0036, 3M Company) was used in the textile studies.

Methods of treating the powder-coated towpreg to improve its "braidability" included towpreg twisting, serving with a polyvinyl alcohol (PVA) monofilament, remelting the powder during braiding and application of surface lubricants such as zinc stearate, hydroxyl-terminated polyethylene glycol (PEG) and polyacrylic acid.

Powder-coated towpreg was twisted 15 twists per meter, a standard level for carbon fiber manufacturers. Earlier studies showed that this twist level had no significant effect on the mechanical properties of woven and unidirectional composites. However, braiding efforts were unsuccessful because twisting did not sufficiently decrease the surface friction of the towpreg.

Serving the powder-coated towpreg with polyvinyl alcohol (PVA) monofilaments in a double helix wrap produced a yarn that was round and tightly compacted. The tightly served yarns braided much like wires or monofilaments - a condition that was not conducive to achieving good lay-down and full coverage of the mandrel.

Surface glazing by re-melting the powder on the surface of the towpreg during the braiding operation was tried. While the glazing treatment reduced, friction, the adhesive properties of the resin caused the tows to stick together and preclude successful braiding.

Application to the towpreg of a small amount (5 percent) of zinc stearate powder was investigated as a means of achieving dry lubrication. In particular, the need for lubrication of towpreg yarns was most apparent when axial yarns are required in the final fabric. This treatment was successful and a tight braid of 10 ends per inch with a 6K towpreg was achieved. The subsequent fabric was processed into composite panels which, unfortunately, were voidy and yielded poor mechanical properties.

One of the major reasons PEG was selected as a lubricant is the fact that it is soluble in and reacts with epoxies. Towpreg, surface coated with it at a level of 1-2 percent, exhibited no significant increase in lubricity. However, at a concentration of 10 percent, the yarn's lubricating quality was significantly increased. Unidirectional composites were made of towpreg coated at 2 and 10 percent levels to evaluate the effect on mechanical properties. Composites made from towpreg containing 10 percent PEG consolidated badly. Their void contents were above 5 percent. The material coated with 2 percent PEG consolidated well and exhibited good interlaminar shear and flexural properties even at elevated temperature.

Surface application of less than 1 percent polyacrylic acid gel provided towpreg with reduced friction. Successful braiding on standard equipment was achieved by first rewinding the gel-coated towpreg onto braiding spools or cops. Since the gel is 99 percent water, most of the water evaporated and the towpreg was dry by the time the braider was loaded with bobbins and the process started. Water was sprayed onto the towpreg yarns once they reached the cross-over region. The lubricating properties of the gel were reactivated with the spray water and a high quality triaxial $[0^\circ/\pm 60^\circ]$ braided fabric with 10-11 ends per inch was obtained using 6K towpreg. The interlaminar shear and flexural properties of composites made with this product were identical to those obtained from control specimens made with untreated towpreg, even at elevated temperatures [3]. While both the PEG and the polyacrylic acid gel enhanced braidability, the latter was preferred because of its ability to be reconstituted with water and the low concentrations required for achieving high lubricity.

With powder-coated textile preforms, a higher resin content is necessary in order to fill all interstitial spaces. However, consideration must be made for the greater amount of debulking

that occurs during consolidation. Consolidation of powder-coated textile structures requires a cure process which will accommodate an approximately 4 to 1 change in thickness of the bulky woven or braided preforms. Previously, multi-step processes were used to fabricate blade stiffened structures made from powder-coated towpreg because of the large amount of debulking that is required.

Recently, the ability of high temperature cast silicone rubber tooling to expand and produce significant pressure at the cure temperature has been demonstrated [4]. A modified silicone rubber system designed to produce accurate pressure application at predetermined temperature levels was combined with movable hard tooling to develop a one step debulking and cure process to fabricate composite structure from textile preforms. Upon visual and microscopic examination, an integrally woven blade stiffened was found to be well-consolidated, wrinkle-free and to have 1 percent voids (Figures 1 and 2).

TOW PLACEMENT RIBBON

Automated tow placement (ATP) is one of the most important new techniques for rapid, cost effective, net shape composite fabrication. The technique has 2 approaches. In one, tacky drapeable thermoset tape is robotically applied to a tool followed by autoclave consolidation/cure. In the other, which the Langley Research Center is emphasizing, a preconsolidated thermoplastic ribbon is thermo-welded on-the-fly. The latter provides in-situ consolidation and obviates the need for autoclave processing, thereby reducing costs. Many commercial research efforts are developing the heated head robotic hardware and associated software to bring this technology into widespread use in building aircraft parts.

As these ATP developmental research efforts succeed and fail, important limitations and "bottle-neck" issues have been discovered. Examples include open section residual stresses, turning radius limitations, autohesion requirements, compliant roller issues, prepreg material quality and post process annealing of crystalline polymers. Most basic of these is the requirement for high quality, fully consolidated, narrow width thermoplastic prepreg ribbon.

Dry powder prepregging of thermoplastics is efficient in distributing solid polymer particles throughout continuous filament tows. The resulting towpreg yarn is flexible, bulky and abrasive. Robotic placement material handling systems are generally designed to utilize stiff, preconsolidated ribbons with consistent cross-section. Research was initiated to develop a bench-scale processing method to convert 1 or 2 powder-coated towpreg yarns into a fully preconsolidated narrow width ribbon acceptable for ATP studies [5]. A comprehensive study of debulking techniques revealed a variety of issues critical to effective ribbonizing including towpreg material quality, transverse squeeze flow, appropriate timing for heating and pressure application and tool contact/release.

Several processing techniques were designed, built and experimentally evaluated to serve as a basis for understanding the unique characteristics of the towpreg ribbonizing process. Use of reactive plasticizers or solvents was excluded from this study. Three powder towpreg yarn materials, Aurum™-500/IM-8 (prepregged by CYTEC), LARC™-IA/IM-7 and PEEK/AS-4 (prepregged by NASA LaRC), were used in the evaluation of these processes. By utilizing desirable attributes of several of the experimental processes, a novel processing technique was developed (Figure 3). The work concentrated on the fabrication of a 0.63 cm wide ribbon from two 12K IM-7 powder-coated tows.

This powder coated towpreg ribbonizer was comprised of two primary components. The hot bar fixture facilitated transverse melt squeeze flow while the cool nip-roller assembly solidified the ribbon into a preconsolidated ribbon with consistent cross-section. The heat transfer and pulling force were modeled from fundamental principles to develop a basic understanding of the process

and help adapt it to a variety of polymeric materials and cure situations. The process has provided quality ribbon from a variety of other high-temperature thermoplastic powder-coated towpreg yarns. The observed experimental rates and temperature ranges indicate that this technique could be readily integrated as a final step in the powder prepreg manufacturing process.

CONCLUDING REMARKS

Significant progress has been made in preparing near net shape parts from powder-coated preforms. The key issues addressed include powder loss during weaving, tow/tow friction during braiding and preform debulking. To reduce losses, the powder was fused to the fiber prior to weaving. Braiding difficulties were overcome by applying a water-based gel to the towpreg. A 4:1 debulking of a complex 3-D through-the-thickness woven blade-stiffened preform was achieved in one experiment in 2 steps; in another, a single step method utilizing expansion rubber molding was used.

A process was developed for the fabrication of ribbon having good dimensional integrity and low voids and useful for heated head ATP studies. In order to implement process control and ensure high quality ribbon, the heat transfer and pulling force were modeled from fundamental principles. These new ribbons, fabricated mostly from dry polyarylene ether and polyimide powders, are currently undergoing in-situ lay-down and evaluation. Quality ribbon is critical to the future development of the hardware for this new technology.

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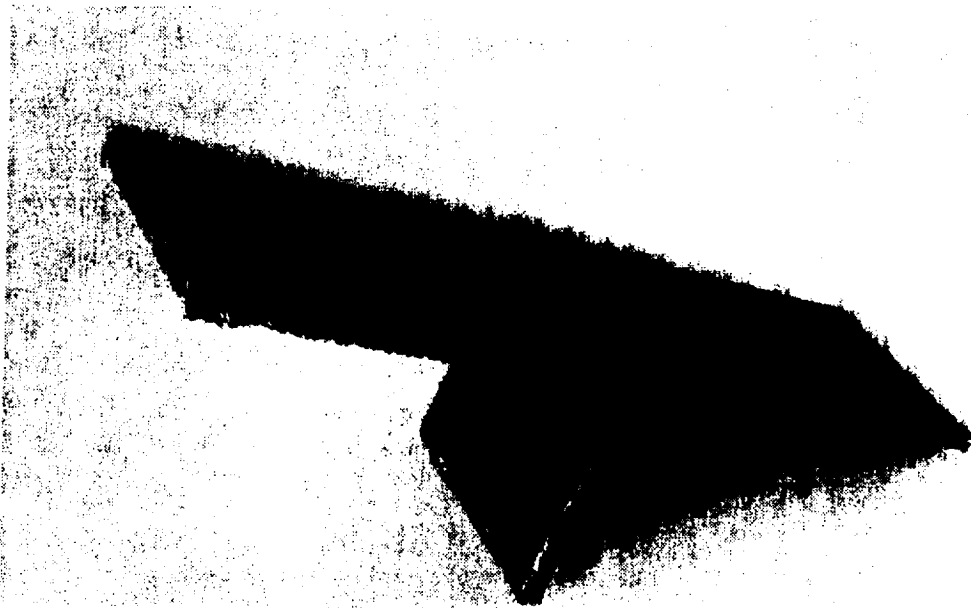


Figure 1. Photograph of the as-fabricated, rubber expansion molded blade stiffener with flashing on outer edges.

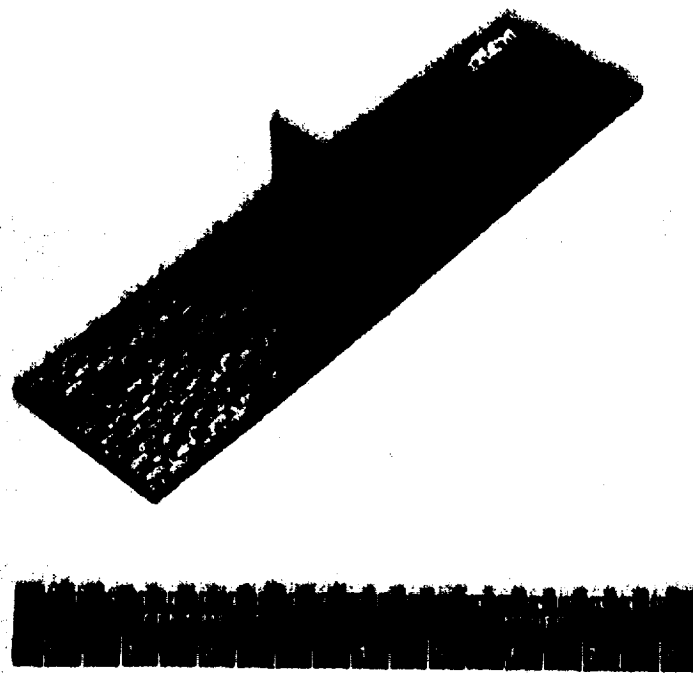


Figure 2. Photograph of the fully consolidated blade stiffener fabricated by the one step process after machining has been performed.

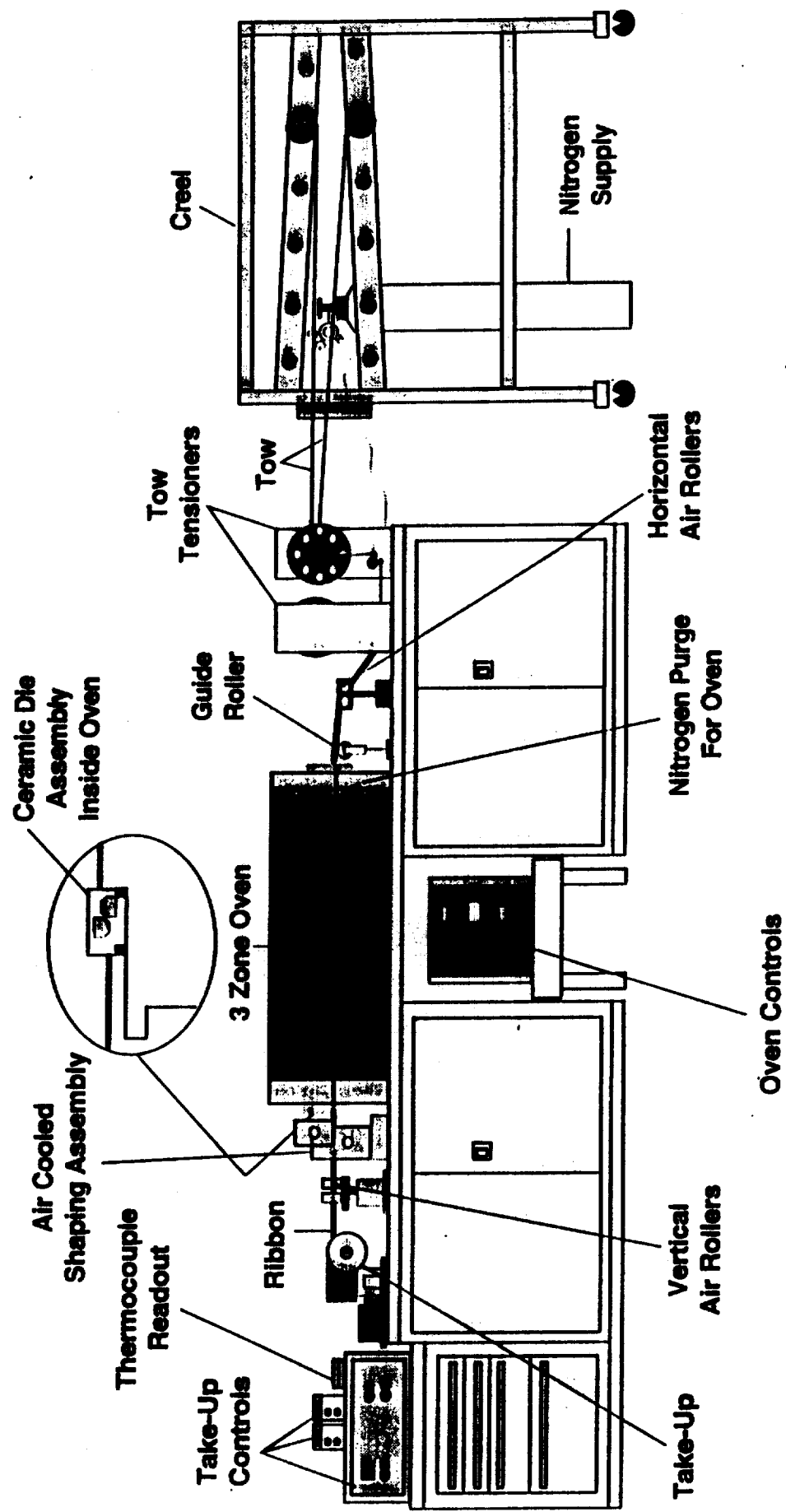


Figure 3. NASA LaRC Powder Coated Towpreg Ribbonizer

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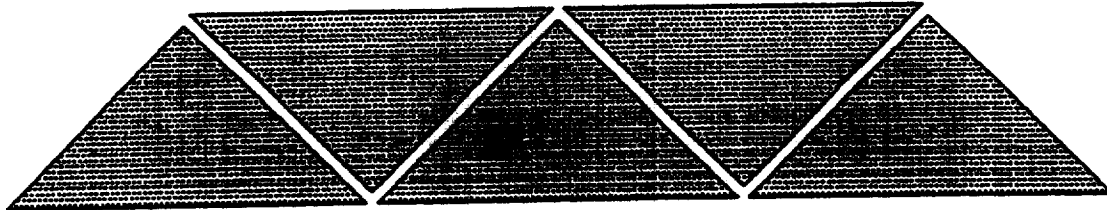
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United States Patent [19]**Sandusky et al.**[11] **Patent Number:** **5,405,683**[45] **Date of Patent:** **Apr. 11, 1995**[54] **NON-RECTANGULAR TOWPREG
ARCHITECTURES**[75] **Inventors:** Donald A. Sandusky, Williamsburg;
Joseph M. Marchello, Hampton;
Robert M. Bancom, Newport News,
all of Va.[73] **Assignee:** The United States of America as
represented by the Administrator of
the National Aeronautics and Space
Administration, Washington, D.C.[21] **Appl. No.:** 141,293[22] **Filed:** Oct. 20, 1993[51] **Int. Cl.⁶** B32B 9/00[52] **U.S. Cl.** 428/225; 428/102;
428/105; 428/232; 428/235; 428/286; 428/294;
428/295[58] **Field of Search** 428/225, 295, 235, 902,
428/102, 105, 286, 232, 294[56] **References Cited****U.S. PATENT DOCUMENTS**

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8.*Primary Examiner*—Patrick J. Ryan*Assistant Examiner*—Abraham Bahta*Attorney, Agent, or Firm*—George F. Helfrich; Joy L.
Bryant[57] **ABSTRACT**

A shaped towpreg ribbon having a cross-sectional geometry which promotes intimate lateral contact between adjacent composite tows was prepared. The cross-sectional geometry is non-rectangular and promotes intimate lateral contact between adjacent towpreg ribbons during normal processing.

3 Claims, 2 Drawing Sheets

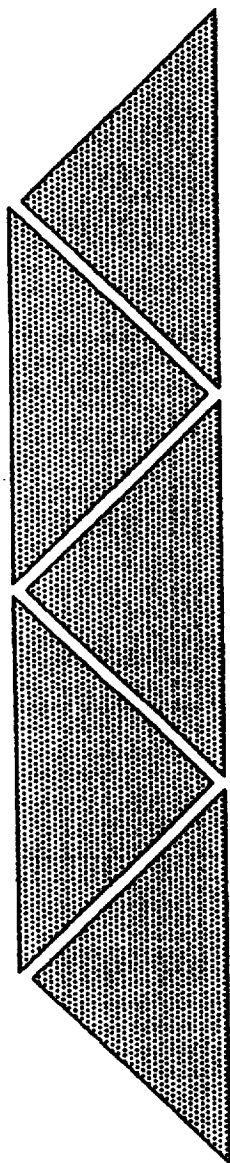


FIG. 1A

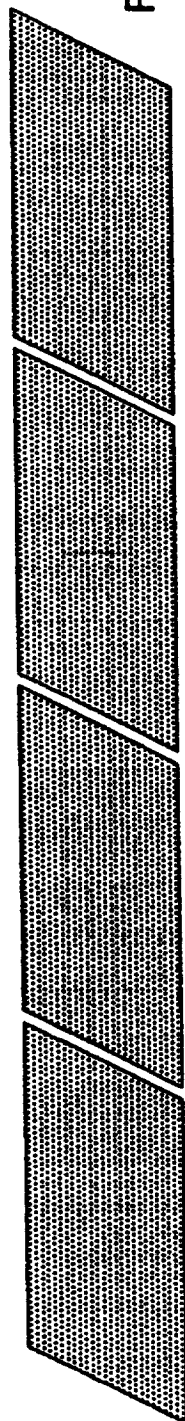


FIG. 1B

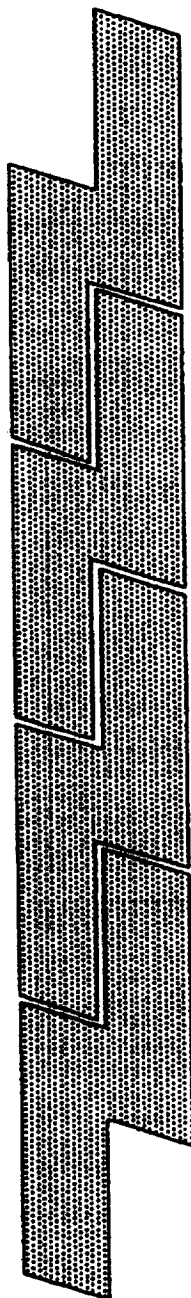


FIG. 1C

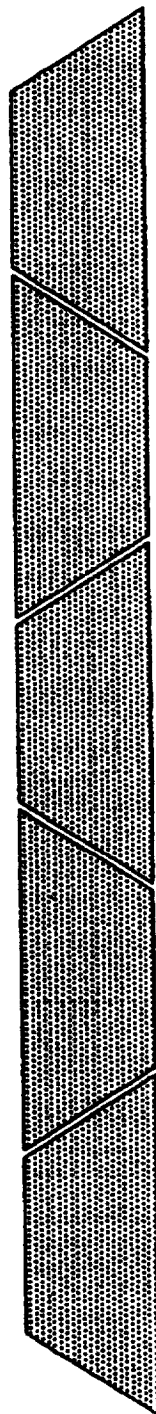


FIG. 1D

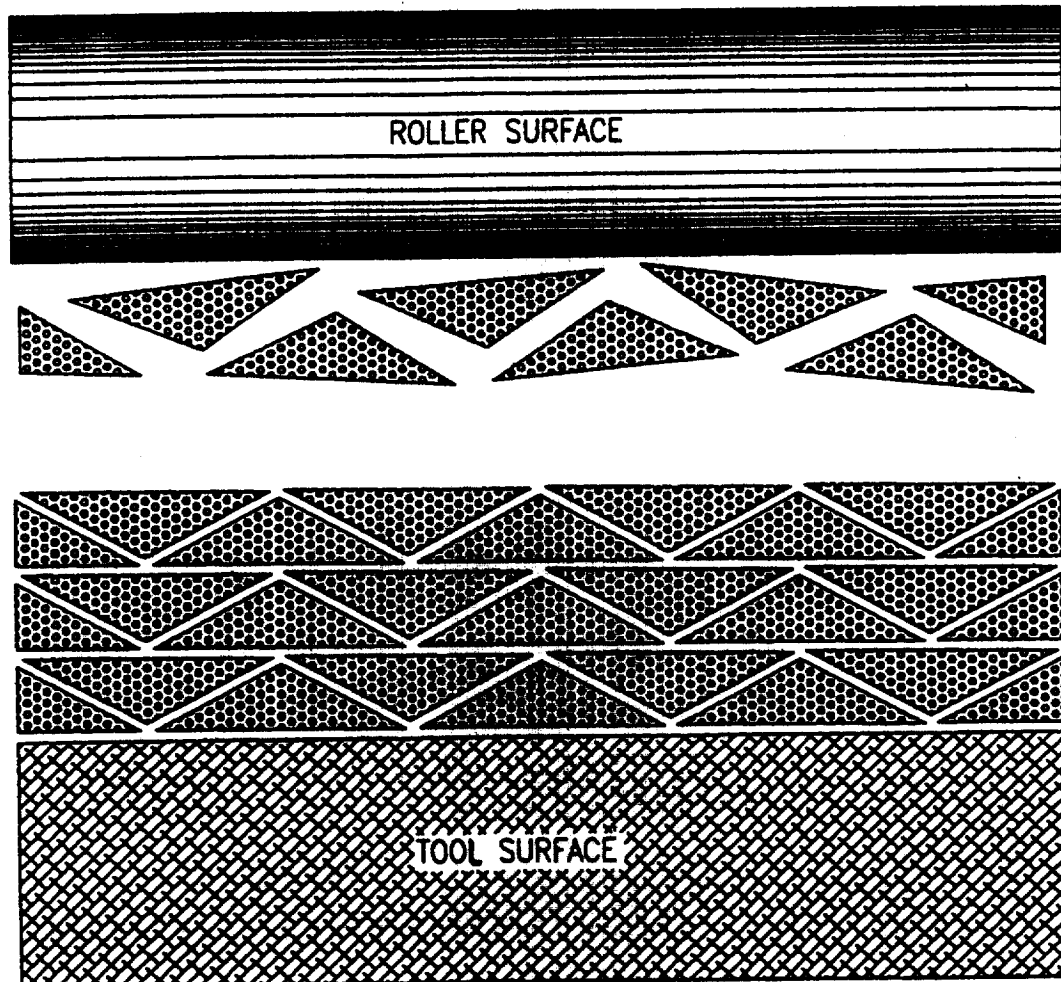


FIG. 2

NON-RECTANGULAR TOWPREG ARCHITECTURES

ORIGIN OF THE INVENTION

The invention described herein was made by an employee of the United States Government and two contract employees in the performance of work under NASA Grant Numbers NGT 51008 and NAG 1067 and is subject to the provisions of Public Law 96-517 (35 U.S.C. 202) in which the contractors have elected not to retain title.

CROSS-REFERENCE TO RELATED APPLICATIONS

This application is related to co-pending patent application Ser. No. 08/141,292, filed Oct. 20, 1993, entitled "Apparatus for Providing a Uniform, Consolidated, Unidirectional, Continuous, Fiber-Reinforced Polymeric Material and Method Relating Thereto".

BACKGROUND OF THE INVENTION

1. Field of the Invention

The present invention relates generally to prepreg ribbons. In particular, it relates to prepreg ribbon forms which have non-rectangular architectures.

2. Description of the Related Art

Ribbonized prepreg is used in the automated placement process where ribbons are robotically managed and continually fed onto a tool or part surface and adhered by application of heat and pressure. This automated placement process is particularly sensitive to ribbon material quality when considering low-flow matrix materials. The simultaneous assembly of adjacent ribbons (typically 4 to 34) offers significant advances in the lay-up of composite prepregs. The low-flow matrix materials (which are often high performance) combined with lack of ribbon cross-sectional dimensional integrity, and most importantly, the existence of the standard rectangular cross-section, complicates the automated placement process frequently rendering poor results. Although ribbons are bonded to their vertical neighbor (directly below) satisfactorily, the failure to make quality parts is generally attributed to the poor bonding of adjacent ribbons to each other. Low-flow thermoplastic parts made by using slit prepreg tapes are typically unconsolidated and exhibit excessive porosity and void content. High to moderate-flow matrix prepreg tapes and ribbons generally provide well consolidated, void-free parts. This contrast can be attributed to the correcting melt-flow nature of the high-flow matrix which allows for simultaneous congregation and filament intermingling of neighboring ribbons.

An object of the present invention is to provide a prepreg ribbon having a cross-sectional geometry which promotes intimate lateral contact between adjacent composite tows.

Another object of the present invention is to prepare a composite from the prepreg ribbons having the aforementioned geometry.

SUMMARY OF THE INVENTION

The objects of the present invention were met by providing a prepreg ribbon having a cross-sectional geometry which promotes intimate lateral contact or nesting between adjacent tows. The cross-section has a plane of contact which provides intimacy of joining upon normal processing. Examples of such cross-section include a triangle, a trapezoid, a parallelogram, and a polygon having more than 4 sides. Essentially, any shape will meet the objectives of the invention as long as it is not rectangular or circular and allows for nesting of the ribbons upon consolidation.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1A is a diagram of the triangular cross-sectional geometry.

FIG. 1B is a diagram of the parallelogram cross-sectional geometry.

FIG. 1C is a diagram of the cross-sectional geometry for a polygon having more than 4 sides.

FIG. 1D is a diagram of the trapezoidal cross-sectional geometry.

FIG. 2 is a diagram of the grouping and alignment of the ribbons.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

As a measure of the capabilities of the current technology, a low-flow hot-melt prepreg tape was slit into 1/4 inch rectangular ribbons and robotically placed onto a tool surface. Following a robotic in-situ manufacturing process known to those skilled in the art, a flat panel section was produced. The panel was placed into an autoclave and processed under vacuum, pressure and heat. The resulting panel contained adjacent ribbon overlaps and gaps. Mechanical testing proved this part was unacceptable.

A low-flow, 12 K, powder coated towpreg was heated and shaped into a non-rectangular cross-sectional architecture. This was achieved by continuously pulling the powder coated towpreg through a forming means wherein the towpreg was consolidated and pre-shaped into a wide, flat, cross-sectional form. Upon exiting the forming means, the towpreg was consolidated into a malleable, wide, flat, cross-sectional form and passed into a shaping means. The shaping means reshaped the malleable, consolidated, flattened polymeric prepreg material into a towpreg having a non-rectangular cross-sectional architecture. Utilizing the same process as that which was disclosed for the slit tape, a composite part was fabricated. The part was well consolidated.

What is claimed as new and desired to be secured by Letters Patent of the United States is:

1. A shaped towpreg ribbon having a cross-sectional geometry which promotes intimate lateral contact between adjacent towpreg ribbons having a similar cross-sectional geometry wherein the cross-sectional geometry is selected from the group consisting of: a triangle, a trapezoid, a parallelogram, and a polygon having more than 4 sides.

2. The shaped towpreg ribbon according to claim 1, wherein the cross-sectional geometry is a triangle.

3. A composite prepared from the shaped towpreg ribbon according to claim 1.

* * * * *